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Cationization of Cotton Using Extracted Keratin from Ethiopian Sheep Wool Waste for Salt Free Dyeing with Reactive Dye

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Abstract

Conventionally, dyeing of cotton with reactive dye can be done in the presence of salt to improve exhaustion by reduction the repletion forces in between the anionic dye and hydroxyl functional groups of cotton. But after dyeing salt and hydrolysed reactive dye effluent becomes directly disposed to the environment and critically affect human health and biodiversity live inside water bodies. To eliminate salt consumption and enhance wool waste utilization, the present study focused on an eco-friendly approach of salt free dyeing of cotton with reactive through cationization using extracted keratin hydrolysed from Ethiopian sheep wool waste. Keratin protein was successfully extracted from wool waste using different combinations of NaOH Concentration, Temperature, pH and Time. The optimum extraction parameters were selected by investigating the maximum absorption obtained at λ max under UV/Vis Spectrophotometer. The dyeing efficiency of keratin treated cotton was compared with untreated cotton. The dye bath exhaustion percentage for cationized by pad-dry, pad –dry-cur and untreated cotton fabric was evaluated using UV/Vis spectrophotometer and recorded as 70 %, 63.3% and 56.6% respectively. The chemical composition of the cationized fabric was investigated under FTIR. The color strength (K/S), CIE L*a*b* was examined under Color eye - 300 spectrophotometer and the cationized fabric shows better K/S value as compared with untreated fabric. The color fastness for both cationized and untreated dyed fabrics also evaluated and investigated using international standards. The cationized cotton fabric shows very good-excellent color fastness property which is better than that of untreated fabric.

Keywords: Cationization; Keratin extraction; Cotton fabric; Salt free dyeing; Reactive dyes; Exhaustion

Introduction

In industrial process, reactive dye was widely used for cotton fabric dyeing, it has wide color gamut, producing brilliant and high wet fastness [1,2]. Reactive dyes stand out from other dyes by their ability to make covalent bonds between carbon atoms of dye reactive group and oxygen atoms of cotton hydroxyl groups under alkaline conditions.

Cellulosic fibers when come in contact with water produce slightly negative charge due to the ionization of hydroxyl groups, whereas most of the dye classes suitable for cotton are anionic in solution. The slightly negative charge on the fiber results in repulsion of anionic dyestuffs and the exhaustion of the bath is limited [3]. However by adding an electrolyte like sodium chloride or sodium sulphate, the charge repulsion factor can be offset and those increased dye exhaustion is achieve [4,5].

The reactive dyes, in particular, required large quantities of electrolyte for its exhaust application, leading to environmental problems. In addition inadequate dye bath exhaustion and dye fixation pose the problem of color effluents [6-8]. After dyeing, these electrolytes are neither exhausted nor destroyed and only 60-65% dye utilization is attainable [9]. The residual dyes and electrolyte was discharged as effluents have caused severe environmental problem and disorders in living organisms [10].

Higher electrolyte concentration in the effluent causes worst effect such as; impairing the delicate biochemistry of aquatic organism, destructive attack on pipes if sodium sulphate is used as electrolyte due to the formation of alumino-sulphate complexes which swell and crack concretes with considerable alumina content. This may lead to emission of hydrogen sulphide gas under anaerobic conditions, dissolution of such sulfides and subsequent bacterial oxidation, which may form the corrosive sulphuric acid. The aforementioned process will lead to higher Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD) and Total dissolved Solid (TDS) [11]. Finally, if the effluent must be treated, i.e. desalinated, the additional cost of the process renders desalination unattractive just from an economical point of view.

Therefore, an alternative approach to eliminate or reduce on salt consumption and improve dye utilization and minimize environmental problems is important.

Having this in mind, The aim of this paper is to study the use of Ethiopian sheep wool waste extracted keratin Hydrolysate as a sustainable material, which can be used in the cationization of cotton during the dyeing of cotton using reactive dyes.

Keratins are difficult to degrade by the common proteolytic enzymes and their disposal leads to environmental problems [12].

The main component of wool protein, which is 95% of the total mass is considered as a useless waste material in most parts of the world and its accumulation in waste streams causes many environmental problems [13,14]. Due to slow degradation, it stays in the dumps/ waste streams for long occupying large volumes of space. Over time, the accumulated hair increases the nitrogen concentration in the water bodies, causing problems of on aquatic life. Open dumps of hair generate hair dust which causes discomfort to people near them and, if inhaled in large amounts, can result in several respiratory problems. The

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best way to address such problems is to develop systems which utilize the waste material as a resource. Therefore, from both an economic and environmental point of view, it is quite desirable to develop an effective and profitable process to use these kinds of resources. As a potential material resource, wool waste has the advantage that it is completely biodegradable, renewable, and available in every locality.

If the waste could be used as a valuable resource, it could not only turn waste to treasure, but also reduce environmental pollution. This has been reported in many studies in relation to the application of other waste. However, no one has conducted a study on the use waste of Ethiopian sheep wool keratin as a cationizing agent for cotton. From theoretical considerations Ethiopian sheep wool keratin will have good reactive properties due to the presence of a large number of reactive amino hydrophilic polar groups (nucleophilic groups) within its molecular structures. If it is possible to synthesize a kind of protein derivative agent, the agent can be applied to cotton, and hence enable salt-free dyeing of cotton using reactive dyes. Such an attempt will lead to use of locally available wool waste bio product as source of keratin hydrolysed to cationized cotton for salt free dyeing. Three advantages will overcome behind this research. One, the environment will be protected from accumulation of the wool wastes disposed from local butcheries and leather tannery industries. Secondly, the dyeing process will be able to reduce electrolytes in the dyeing water effluents. Third, there will be a probability of building strong covalent and ionic bond in between the cationic functional groups of treated cotton fabric and the anionic reactive dye molecules leads to enhance the color fastness properties of the dyed fabric. Those three advantages will be a welcomed by the advocates of greener production.

Experimental

Materials

Waste of wool was collected from Dire Dawa City local butcheries and leather tannery industry. Full bleached 100% cotton fabric with count 21 Ne warp and weft count with 74 ends per inch and 66 picks per inch was obtained from Ethiopian Institute of Textile and Fashion Technology (EiTEX) Laboratory.

Chemicals and auxiliaries: Sodium hydroxide (NaOH), Sodium carbonate ($NaCO_3$), Sodium chloride (NaCl), Acetic acid (CH_3COOH), Blue DCT reactive dye, detergent and water were used throughout this research work to carry out keratin extraction, cationization and dyeing processes. All those listed chemicals auxiliaries were obtained from Ethiopian Institute of Textile and Fashion Technology (EiTEX) Laboratory.

Equipment and apparatus: Digital electronic balance, The pH meter, Perkin Elmer UV/VIS Spectrometer, COLOR-EYE 3100, FT-IR Spectroscopy Perkin Elmer, wash fastness tester, light fastness tester, rubbing fastness tester, auto dryer, Infrared dyeing machine, padder, Electrophoresis were mainly used throughout this research work and found from Bahir Dar University, Ethiopian Institute of Textile and Fashion Technology (EiTEX) and Ethiopian leather Industry Development Institute (LIDI).

Methods

Extraction of keratin hydrolysate: Wool was collected manually washed thoroughly with hot water and detergent to remove impurities and dirt on the surface of human hair. The cleaned hair was exposed to sunlight for drying and then converted into small particles manually using scissors.

Keratin was extracted from wool by immersing in 40 g/l of NaOH solution at pH 12 to 13 for 2 to 5 hrs [15]. Wool keratin was extracted by hydrolysis with 3% (O.W.M) of NaOH at boil for duration for 6 hours.

In this excrement nine Extraction trials of experiments were conducted. Before dissolution, wool waste was intensively washed with fresh water, dried and cut into small pieces. 50 gram of wool fiber was extracted at different combinations of Temperature (40, 60 and 80°C), pH levels of (13, 12 and 11), Time (1, 2 and 3 Hr.) and Concentration of NaOH (20, 30 and 40) g/l respectively. Parameters combination was scientifically done by using Minitab software.

The resultant solution was filtered and its absorption was determined by UV-Visible spectroscopy. The temperature, concentration, pH and time combination yielding the maximum absorption were taken as optimum condition for extraction of keratin from human hair.

Cationization: The extracted keratin Hydrolysate was purified using electrophoresis method and then applied on full bleached cotton fabric by pad-dry and pad-dry-cur techniques. The cationized and control (untreated) cotton fabrics were dyed by exhaust method.

Evaluation of FTIR spectrum: The chemical of cationized and untreated cotton fabrics were evaluated using FTIR.

Dyeing procedure for cationized and untreated cotton fabrics: Exhaust dyeing method was followed using Infrared (IR) dyeing machine for both treated and untreated cotton fabrics. The dyeing parameters were followed as per the conventional dyeing of cotton fabric with DCT reactive dye except the cationized fabric was dyed without salt whereas dyeing of untreated cotton fabric was done in the presence of salt as shown in Figures 1 and 2.

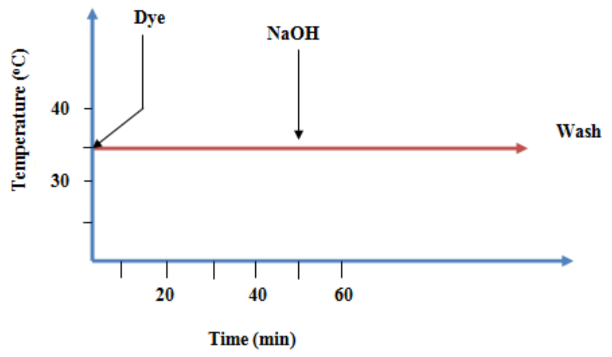

Figure 1: Typical dyeing cycle with Dichlorotriazinyl (DCT) reactive dye for cationized cotton fabric.

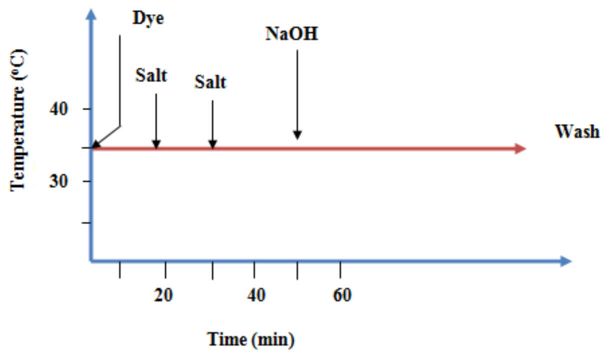

Figure 2: Typical dyeing cycle for untreated cotton fabric with Dichlorotriazinyl (DCT) reactive dye.

The cationized cotton fabric with wool waste extracted keratin was immersed in 1% (o.w.f, weight percent of dye relative to fiber) of DCT reactive dye solution with a liquor ratio of 1:20. The dyeing temperature was kept at 30ºC constantly for 60 min and the pH of the dye solution maintained at 11. Later, 5 g/L Na_2CO_3 was added to the dye solution after 45 min and continue dyeing at 30ºC for 15 min. Dyed fabric was rinsed with cold water and finally washed with hot water using detergent at room temperature and allowed to air dry.

The untreated cotton fabric was immersed in 1% (o.w.f) of DCT reactive dye solution with a liquor ratio of 1:20 and total dyeing time of 60 min. The dyeing temperature was kept at 30°C for 15 min before salt addition (initial exhaustion) and then 30 g/L of NaCl was gradually added to the dye bath with basis of 15 min intervals. Then 5 g/L Na_2CO_3 was added to the dye bath after 45 min at 30°C and then continue dyeing for 15 min after Na_2CO_3 was added.

After dyeing, the dyed fabrics was rinsed with cold water and finally washed with hot water using detergent.

Evaluation of dye uptake

Dye exhaustion: The percentage dye exhaustion for DCT reactive dye was determined by comparing concentration of dye in dye bath before and after dyeing. This was done with the help of a UV/Vis spectrophotometer at the maximum wavelength of absorbency and a calibration curve. The exhaustion is calculated using equation:

Determination of dye exhaustion (E%)

$$E\% = \frac{(A_0 - A_1)}{A_0} \times 100$$

Where, Ao and A1 indicate the absorbencies at maximum wavelength (λ_{max}) of the dye originally in the dye bath and of residual dye after dyeing respectively.

Determination of color measurement: The color strength (K/S), Reflectance percentage (R%) and CIEL*a*b* values of dyed fabrics were evaluated using color eye-300 software based on D65 illuminants and 10o observer. The reflectance (R) value of dyed cloth at maximum wavelength of absorbency (λ max) is found and the K/S was calculated by using the formula stated below.

$$K/S = \frac{(1-R)^2}{2R}$$

Where, K/S and R stands to color strength and reflectance at maximum wave length (λ_{max}) respectively.

Determination of color fastness: Color fastness such as wash fastness, light fastness and rubbing fatness properties of the dyed cotton both treated and untreated fabrics were determined as per the international testing standards.

Result and Discussion

Keratin extraction optimization

Wool Hair was collected from local butcheries and then washed using detergent and hot water to remove the dirty and oil substances. After washing it was dried at room temperature and chopped in to small particles using scissors to facilitate dissolution. 50 g keratin was extracted from wool hair/fiber waste by conducting 9 experimental trials with different extraction parameters (pH-11, 12 and 13), (Temperature-40, 60 and 80°C), (Time 1, 2 and 3 Hrs.), (Concentration of NaOH-20, 30 and 40 (g/l)).

The extraction was done in a closed Infrared Dyeing Machine with computer aided parameter controlling mechanism. The absorption of extracted keratin Hydrolysate of each trails were evaluated using UV-Visible spectroscopy (Lambda 25). The maximum Absorption was obtained from trial 5 with absorption of (4.0) at λ_{max} 260 nm. Therefore, Concentration of 40 g/l of NaOH, Temperature of 80°C , and Time of 3 Hours and pH of 13 was optimum condition for keratin extraction from wool hair (Table 1 and Figures 3 and 4).

Effect of time, PH, NaOH concentration and temperature on yield % and UV absorption of extracted keratin from wool waste: As extraction Time, concentration of NaOH and pH separately increase and other parameters kept constant, the yield % and absorption of extracted wool keratin also increase as showed on Figures 5-10. Similarly, as temperature increase and other parameters kept constant, the absorption/concentration and yield % of extracted wool keratin Hydrolysate also increase as showed on Figure 11.

Cationization of Cotton with extracted keratin from wool waste

50 gram of full bleached cotton fabric was immersed in 50 g/l of keratin haydrolyzate extracted from wool hair and was subjected to drying and dry- cur methods at 80°C , 60°C and 100°C for 4, 2 and 2 minutes respectively. The peak of FTIR spectrum curve of fabric treated and untreated with wool extracted hydrolyzat was showed in Figure 4. The peaks on the FTIR curve showed that there is a change in chemical composition after being cationized. This indicates the keratin hydrolyzat was fixed to the fabric.

The microstructure and chemical composition characterization of micro particles were examined by using FTIR spectroscopy (PerkinElemer) in the absorption mode with the 4000 cm^{-1} and 500 cm^{-1} wave number range. It will help to detect the changes in chemical composition of peptides [16].

MLR: 1:20					
No of Trials	**pH**	**Temperature (ºC)**	**Concentration of NaOH (g/l)**	**Time (hr.)**	**UV Absorption (A %) at λ_{max} (260 nm)**
1	12	60	30	2	2.4
2	11	40	20	1	3.4
3	13	40	40	2	2.7
4	12	80	30	2	3.8
5	13	80	40	3	4.0
6	12	40	20	3	2.5
7	13	40	30	2	2.5
8	13	80	20	3	3.1
9	11	80	20	1	2.8

Table 1: Absorption of extracted keratin from wool waste.

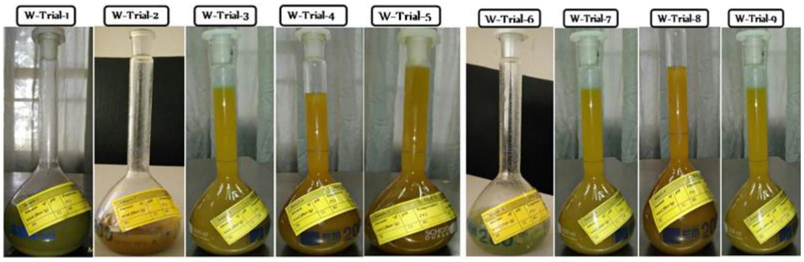

Figure 3: Extracted keratin Hydrolysate from wool waste.

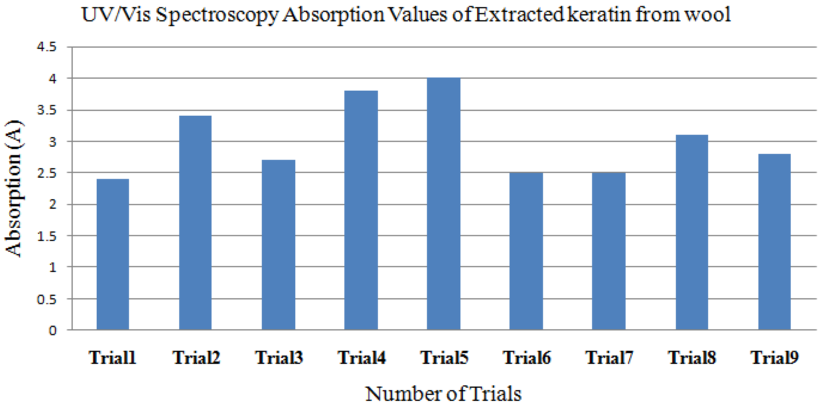

Figure 4: Maximum absorption value for different trials of extracted Keratin from Human Hair at λ_{max} (275 nm).

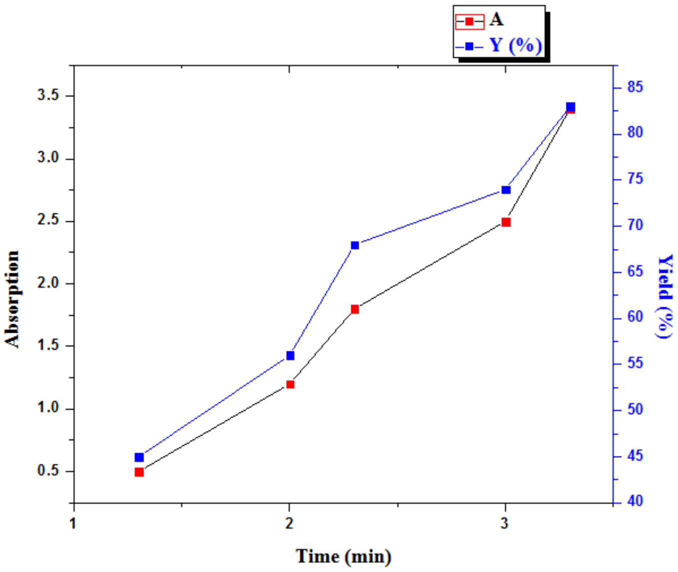

Figure 5: Effect of Time on absorption and yield % of keratin extraction from wool.

The FTIR spectrum of cationized and untreated cotton fabrics were illustrated in Figure 9. Compared with untreated cotton, the absorption peak at about Peaks at 1644 and 1625 cm^{-1} were appeared which was assigned to bending vibration of N-H primary amines. This observation clearly indicated the presence of primary amines which fully demonstrated that the amino group was chemically absorbed on the treated cotton fabrics. It indicated that the reaction between cotton fabrics and cationic keratin polymer occurred. The content of amino groups present on the fabric treated by pad-dry-cur method was higher than that of treated by pad-dry method (Figure 9).

Dyeing of treated and untreated cotton fabric with Dichloro Trazline reactive dye

Dyeing was carried out as per the conventional dyeing procedure at room temperature for 60 min. washing and soaping was done for both treated and untreated cotton fabrics. The visual actual color yield

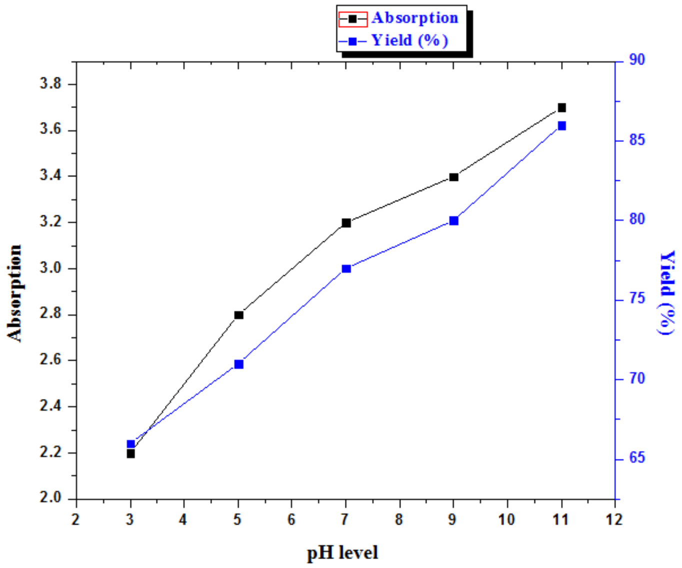

Figure 6: Effect of pH level on absorption and yield % of keratin extraction from wool.

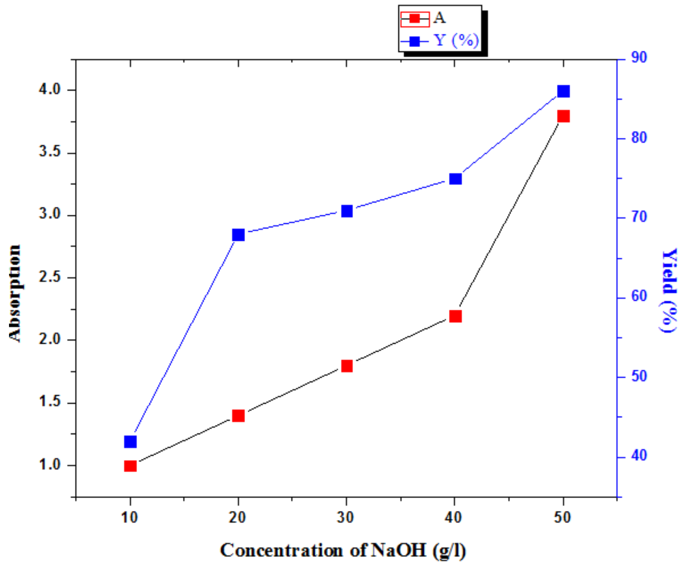

Figure 7: Effect of NaOH concentration on absorption and yield % of keratin extraction from wool.

of treated fabric was higher than that of untreated fabric sample. Even fabric treated by pad-dry-cur method was higher than that of treated by pad-dry method as shown in Figure 10.

Dye exhaustion percentage

The percent exhaustion of DCT reactive dye before and after dyeing of the cationized and untreated fabrics were evaluated and recorded in Figure 11. The dye shows higher percent of dye exhaustion on cationized cotton than untreated cotton sample. Even the dye exhaustion on treated cotton by pad-dry-cur method with (E%=70) was higher than that of cotton treated by pad-dry method with (E%=63.3) and untreated cotton sample (E%=56.6) (Figure 11).

Determination of color strength (K/S) and CIE $L^*a^*b^*$ value

The color strength, reflectance value and CIE $L^*a^*b^*$ Value of dyed cotton samples which are untreated and treated with human hair extracted keratins were measured in the range of 400-700 nm using Color Eye 300 and recorded in Table 2. Color strength (K/S) indicates how much concentration of dye solution was fixed or attached to the cotton fabric after dyeing.

The values used by CIE are called L*, a* and b* indicates, L* (lightness (L=100)/darkens (L=0)), a* (redness (+a)/greenness (-a)), b* (yellowness (+b)/blueness (-b)) value of dyed cotton samples. The lightness (L*) value of untreated fabric was comparatively higher than cationized cotton fabric samples. All dyed samples shows (+a) means all are in the red direction. But the redness of treated sample was higher than untreated cotton fabric sample. Value of b* in all dyed samples of treated and untreated cotton was in –ve b* direction. But the bluness of treated sample was lower than untreated cotton samples.

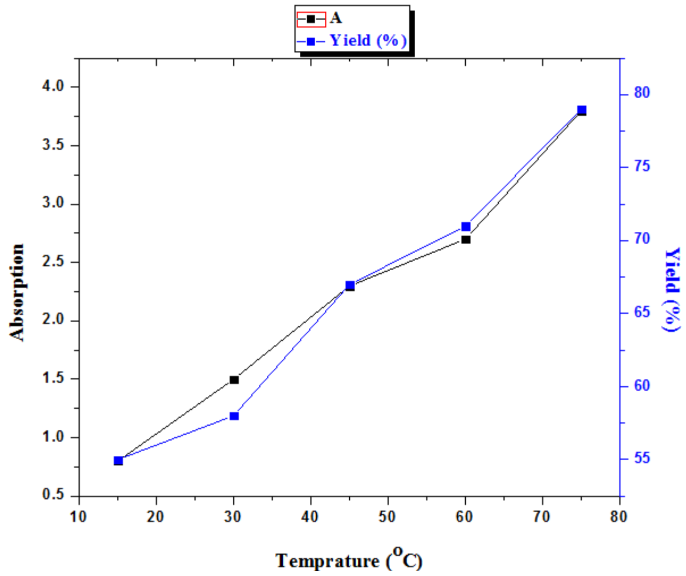

Figure 8: Effect of Temperature on absorption and yield % of keratin extraction from wool.

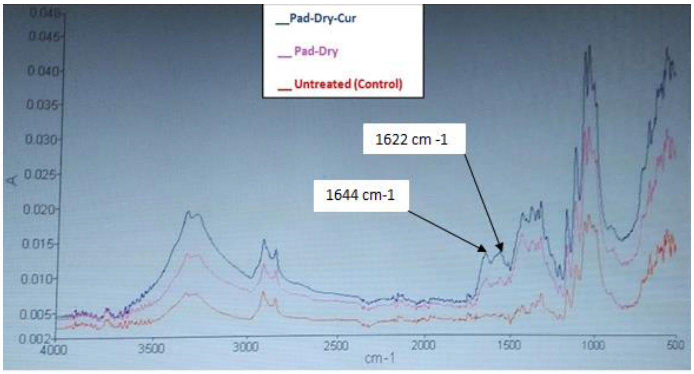

Figure 9: FTIR curve for untreated and treated cotton fabric with wool extracted keratin.

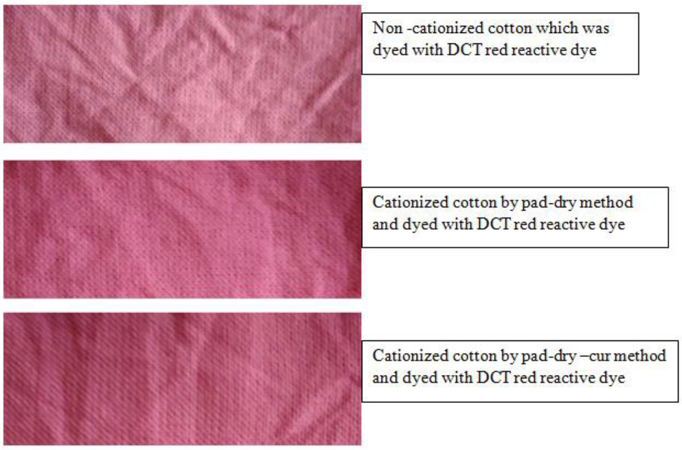

Figure 10: Dyed cotton fabric both treated and untreated by different methods.

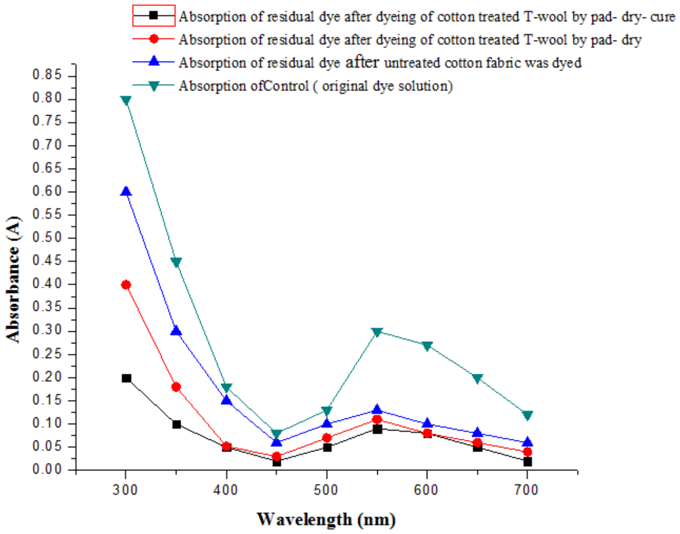

Figure 11: UV/Vis absorbance values of blue DCT reactive dye solution after and before dyeing.

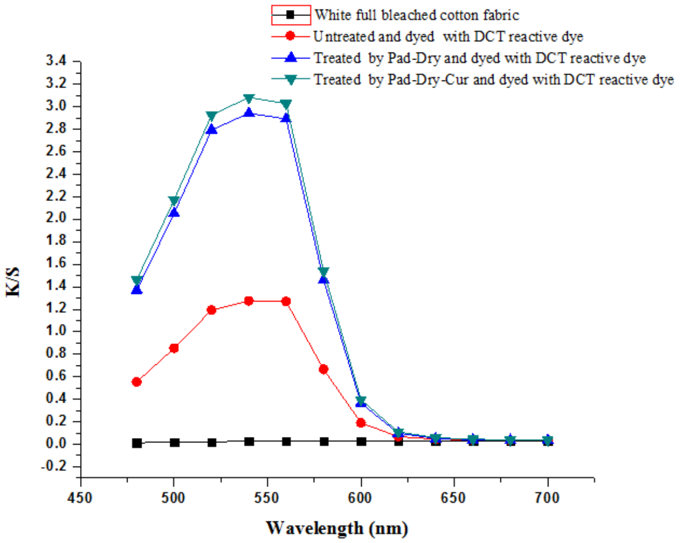

Figure 12: K/S value of untreated and treated with wool extracted keratin and dyed using DCT reactive dye at different wavelengths.

In the exhaust dyeing process, most of reactive dye molecules were easily absorbed and diffused into modified cotton by opposite charges attraction. The absorption could greatly increase the concentration of reactive dyes inside treated cotton, which enhance dye exhaustion and dye -fiber fixation reaction in the dye-fixation process. To some degree, the positive charge of modified cotton could also temporarily restrict the movement of anions, especially the hydroxyl anion. It accordingly decreased the hydrolysis of reactive dyes. So the total utilization of reactive dyes was improved greatly.

The dye ability and color strength (K/S) value of the cationic cotton dyed with DCT reactive dye was higher than that of untreated (conventional) dyed cotton due to the presence of imparted cationic (positive) charge. On the other hand, among the treated cotton fabric sample which was treated by pad-dry-cur method showed comparatively higher K/S value than that of treated sample by pad-dry method and untreated fabric samples as shown in Figures 12 and 13. This indicates the curing temperature during cationization plays grate role on the fixation of more cationic amino functional groups on the fabric leads to take more dye molecules and enhance the color strength of dyed fabric.

In general Cationized fabrics contain positive groups which increase reactive dye exhaustion due to ionic attraction between anionic reactive and cationic (NH^{3+}) of treated cotton fabric. As a result the Exhaustion percentage and K/S value of the cationized fabrics were higher than that of conventional (untreated) fabric (Table 2).

Color Fastness test results of dyed samples

The color fastness such as light fastness, rubbing fastness and washing fastness properties of the dyed fabrics were evaluated based on the international standards of ISO105B02, AATCC 116-1995, ISO 105-A03 respectively.

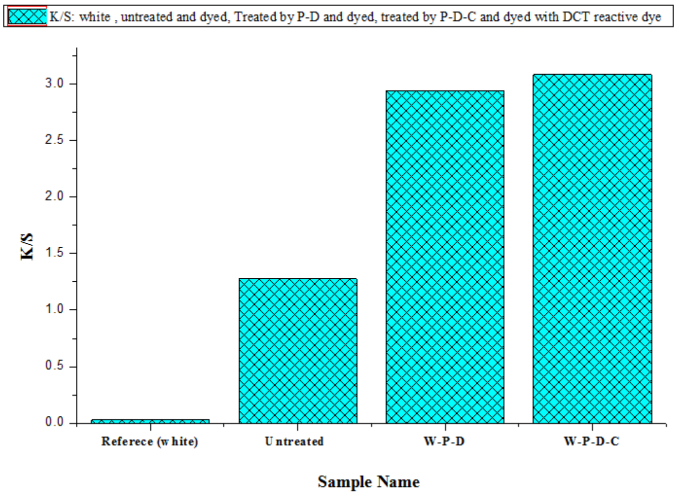

Figure 13: K/S value for white fabric, untreated and treated with wool by pad -dry and pad-dry-cur methods and dyed with DCT reactive dye.

Method	Sample code	Reflectance (%) at 540 nm	K/S at 540 nm	CIE L*a*b* system		
				L*	a*	b*
Cationized with Wool extracted keratin and dyed using DCT reactive red dye	White	78.76	0.0286	91.78	2.28	-9.09
	Untreated	23.15	1.27	65.59	35.79	-14.33
	W-p-d-d	12.87	2.94	56.30	45.22	-9.84
	W-p-d-c-d	12.43	3.08	55.52	44.94	-9.30

Table 2: Color strength (K/S), Reflectance % and CIEL*a*b* values of cationized cotton fabric with wool keratin Hydrolysate and dyed using DCT reactive dye at (10o observer).

Sample Name	Sample Code	Rubbing fastness		Light fastness	Wash fastness	
		Dry	Wet	24 h	Acidic	Basic
Cationized with Wool extracted keratin and dyed using reactive(DCT) red dye	Untreated	3/4	3/4	4	3	3
	W-p-d-d	3	3	5	3/4	3/4
	W-p-d-c-d	3	3/4	5	4/5	5

Table 3: Color fastness properties of untreated and treated cotton fabric with wool waste extracted keratin and dyed with DCT reactive dye.

Color fastness to washing was assessed in respect of color change and staining on multi-fiber fabric (acetate, cotton, nylon, polyester, acrylic, wool). Wash fatness of the dye would depend on the bonding between the keratin (NH^{3+}) and cellulose. To study this effect the washing fastness of the dyed samples of treated and untreated cotton fabrics were evaluated. The cationized cotton fabric samplers showed lower color reduction after repeatedly washing (5 wash cycles) compared with untreated cotton.

Rubbing fastness was evaluated in dry and wet conditions. Fastness ratings of different types of dyed samples are presented in the.

The table showed that the light and washing fastness ratings of keratin treated fabric shows better performance as compared to untreated fabrics. Due to presence of additional cationic functional groups on the treated fabric, there was a probability to form strong covalent and ionic bonds in between the treated fabric and reactive dye molecules which leads to enhance the washing and light fastness properties as compared to the untreated fabric. However, in case of rubbing fastness both wet and dry conditions, the keratin treated fabrics showed lower rubbing fatness compared with untreated fabrics due to the probability of significant change surface condition occurred after cationization. In general, the performance of color fastness for cationized cotton fabric shows better performance as compared to the untreated fabric. Specially prior to dyeing, fabric which was treated by pad-dry-cur method shows very good to excellent color fastness property as compared to the untreated and even treated by pad-dry method (Table 3).

Key: W-P-D (pad-dry with wool extracted keratin), W-P-D-C (pad-dry-cur with wool extracted keratin).

Conclusion

The present study revealed that application of extracted keratin from wool waste on full bleached cotton fabric for salt free dyeing with reactive dye. The extracted keratin was applied on full bleached cotton fabric by pad-dry and pad-dry-cur methods and the fabric was tested its color fastness, color strength and CIE L*a*b*, dye exhaustion, FTIR spectrum. Dye exhaustion as well as color fastness of the dyed samples was improved after the cationization. Since Ethiopian sheep wool is dead fiber, it is not used as textile fiber and simply disposed to the environment and accumulated in mass which can seriously affect the environment. There was an opportunity and possibility of converting this waste in to valuable purpose such as used as extraction of natural biodegradable cationic agent for cotton fabric to improve the dye ability and dye exhaustion of cotton with reactive dye without addition of any electrolyte (salt). The result of this research gives three main advantages: one, by converting to usable product the waste of wool will be minimized and the environment keep clean. Secondly, the dye ability, percentage of exhaustion and wash fatness of the reactive dye will be improved after treatment which leads to reduce the effluent of untreated dye disposed to the environment. Thirdly, addition of salt in the dye bath during dyeing of cotton with reactive dye was eliminated and protect environment from this effluent. Therefore, finally it is possible to propose that extracted keratin from wool can be used as bio -degradable eco-friendly cationic agent applied on cotton fabric for salt free reactive dyeing.

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